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MACRO-ENCAPSULATION OF HEAT STORAGE
PHASE-CHANGE MATERIALS
for Use in Residential Buildings

Seventh Quarterly Progress Report

March 29, 1978 - June 29, 1978

by

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16. Abstracts <p>Objectives are to assess the feasibility of macro-encapsulated PCM's for residential solar systems, to develop and evaluate such materials. Encapsulant materials under consideration are multilayer flexible plastic films, steel cans, and plastic bottles. Studies of the storage capability of $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ encapsulated in polyethylene bottles, $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in steel cans, and $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}/\text{NH}_4\text{NO}_3$ eutectic, encapsulated in flexible plastic film packages, have been completed in the sub-scale storage test unit. The next PCM to be studied is NH_4Br/urea eutectic in polypropylene bottles.</p>				
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FOREWORD

The work described in this report is an extension of a basic effort already performed under contract NSF-C906. The general objectives are: 1) to assess the technical and economic feasibility of encapsulated phase change materials (PCM's) for storing heat in residential solar energy systems, and 2) to develop and evaluate such encapsulated phase change materials.

The project involves three tasks:

Task 1 - Materials selection, including a limited literature search, selection of candidate phase change materials, and selection and characterization of encapsulating materials.

Task 2 - Procurement of phase-change and encapsulating materials, encapsulation studies, and testing of the encapsulated materials.

Task 3 - Preliminary design and economic evaluation of a residence-sized heat storage sub-system.

Task 1 has been completed, and Task 2 efforts are in progress.

I. ENCAPSULATED PCM'S

The selection of phase change material (PCM) - encapsulant pairs shown in Table I was based primarily on: (1) container-PCM material compatibility studies, (2) container-PCM costs, (3) PCM fusion temperatures, and (4) a need to cover a wide spectrum of container types.

TABLE I

Selection of PCM-Encapsulant Pairs

<u>Phase Change Material</u>	<u>Fusion Temp (°C)</u>	<u>Encapsulant</u>
$\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$	27	High Density Polyethylene Bottle (HDPE)
$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}/\text{NH}_4\text{NO}_3$ Eutectic	50	R-2 Retort Film
$\text{NH}_4\text{Br}/\text{Urea}$ Eutectic	76	Polypropylene Bottle
$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	89	Drawn Steel Aerosol Can

Because of the difficulty in finding a satisfactory encapsulating medium for stearic acid, we decided to replace this PCM in our program with ammonium bromide/urea eutectic. This material has a heat of fusion of 36 cal/g and a density of about 1.8 g/cc. Due to the cracking experienced with high density polyethylene bottles, we decided to use polypropylene bottles, which are more resistant to cracking and to high temperature, for this PCM. Polypropylene bottles filled with $\text{NH}_4\text{Br}/\text{urea}$ composition were subjected to 89 freeze-thaw cycles in an oven, without any apparent degradation of PCM or encapsulant.

II. HEAT STORAGE TEST PROCEDURE: MAGNESIUM NITRATE HEXAHYDRATE/ AMMONIUM NITRATE EUTECTIC

The ASHRAE¹ standard (94-77) for testing thermal storage devices was followed, modified for testing units using phase change materials.² This standard requires that the inlet and outlet temperatures, prior to the start of the test, be at steady state with a constant flow of air through the thermal battery. The test period begins with a step change of 35°C in the inlet air stream. For a PCM thermal battery the 35°C step change symmetrically brackets the fusion temperature, causing the storage medium to undergo a change of phase. Since the magnesium nitrate hexahydrate/ammonium nitrate eutectic freezes at 50°C, the inlet air temperature steps from 32.5°C to 67.5°C during charging and back to 32.5°C during discharge.

Figure 1 shows a temperature-time profile for the inlet and outlet during the test of a near-ideal thermal battery using the latent heat of the eutectic for thermal energy storage. In the charging mode the test ends when the outlet temperature reaches a temperature midway between the freezing point of the eutectic and the inlet temperature, defined as the "maximum charge temperature," 58.75°C. The charge rate at this temperature, or "minimum useful charge rate" is useful in system design work.

Once steady state conditions are achieved, the inlet air temperature is step changed from 67.5°C back to 32.5°C, where it is held constant to generate a discharge profile of the outlet temperature. As in the charging cycle, the test ends

¹American Society of Heating, Refrigeration and Air Conditioning Engineers.

²ORO/5217-3.

Figure 1

LATENT HEAT TYPE THERMAL STORAGE UNIT

when the outlet temperature reaches the "minimum discharge temperature" (41.25°C), midway between the inlet and freezing temperatures.

The areas between the inlet and outlet temperature profiles over the test times of charging (τ_c) and discharging (τ_d) are used to determine the charge and discharge capacities, CC and DC respectively, using the following equations:

$$CC = \dot{m} C_{tf} \left(\int_0^{\tau_c} (T_{in} - T_{out}) dt - L \int_0^{\tau_c} \left(\frac{T_{in} + T_{out}}{2} - T_a \right) dt \right) \quad (1)$$

$$DC = \dot{m} C_{tf} \int_0^{\tau_d} (T_{out} - T_{in}) dt \quad (2)$$

The second part of equation (1) is the heat loss from the storage unit during the charging cycle. This heat loss calculation, a modified form of the equation given in ASHRAE (94-77), is more accurate since it does not assume a constant temperature driving force between the storage unit and ambient air temperatures ($\bar{T}_{unit} - \bar{T}_a$). The heat loss factor is determined according to the following equation:

$$L = \frac{(T_{in} - T_{out})}{[(T_{in} + T_{out}) 0.5 - T_a]} \quad (3)$$

The temperatures are measured after the unit has achieved uniform steady state temperatures.

III. HEAT STORAGE TEST RESULTS

The thermal energy storage unit used is shown in Figure 2. Pouches were fabricated from R-2 retort film (3.3 mil polyethylene, 0.35 mil aluminum foil, and 0.5 mil polyester), and filled with the $Mg(NO_3)_2 \cdot 6H_2O / NH_4NO_3$ eutectic.

Nomenclature

C_{tf}	=	specific heat of transfer fluid - $\frac{KJ}{Kg^{\circ}C}$
CC	=	charge capacity of thermal storage unit - KJ
DC	=	discharge capacity of thermal storage unit - KJ
L	=	heat loss factor - dimensionless
$\dot{m}$	=	mass flow rate of transfer fluid - $\frac{KG}{HR}$
ΔP	=	pressure differential across storage unit - in. H ₂ O
q	=	heat rate - $\frac{KJ}{HR}$
Q	=	total heat - KJ
Q_{in}	=	total heat delivered to storage unit through transfer fluid - KJ
Q_{out}	=	total heat delivered by storage unit through transfer fluid - KJ
T_a	=	ambient temperature - °C
T_{in}	=	temperature of transfer fluid entering storage unit - °C
T_{out}	=	temperature of transfer fluid leaving storage unit - °C
τ_c	=	charging test time (hr)
τ_d	=	discharging test time (hr)

Figure 2

MAGNESIUM NITRATE HEXAHYDRATE/AMMONIUM NITRATE EUTETIC
STORAGE MEDIUM AIR-HEAT TRANSFER FLUID,
THERMAL ENERGY STORAGE UNIT

Three-seam pouches (seams on one side and both ends) were used, because they survived thermal cycling better than four-seam pouches (seams on both sides and both ends). The pouches were made using a Vertrod® impulse heat sealer (Model 20P-1/8" wide seal). The film was folded lengthwise and sealed on three sides, leaving a small filling section. After the pouch was filled with liquid eutectic, excess air was squeezed out of the pouch and the fill-hole was heat-sealed closed.

Pouches were laid out on metal grills with their seams projecting horizontally (see Figure 2), and emplaced in the test storage unit. Nine thermocouples were glued between half-filled pouches and positioned in the subscale storage unit.

The temperature-time profiles obtained in testing this configuration resemble strongly earlier results for calcium chloride hexahydrate and magnesium nitrate hexahydrate. The profiles are not shown here because after the experiment some of the thermocouples were found to be rusted open.

Several pouches developed split seams, leaking a portion of their contents. Several more showed signs of delamination of the heat seal in the mid-region of the long seam. Of the 75 pouches in the test unit, 22 split and 15 showed signs of delamination after sixteen cycles. In comparison, other three-seam pouches logged 180 cycles in an oven without a failure. The reason for the high failure rate in the test device can be attributed to using a higher % fill. The quantity of eutectic leaked was small, and should have little effect on the test results.

A total of eight pairs of charge-discharge cycles (Runs 9 through 16) were evaluated at four air flow rates (59 through 89 cubic feet per minute, CFM). Profiles for the inlet and outlet air temperatures are shown in Figure 3 for the charging cycles and Figure 4 for the discharge cycles. The profiles are shown only for the test duration. Steady state was achieved before initiating the next step change.

The stipled areas between the outlet and inlet air temperature (shown only for the 89 CFM tests) was integrated numerically and used to determine the capacity. In each cycle it took significantly longer to charge the storage unit than to discharge it. The primary sources of this difference are heat loss from the system during the test, and the lower specific heat and density of the air during the charging cycle. The heat loss effect is approximately twice that of the variation in heat transfer fluid properties. In a scaled-up system with relatively smaller heat loss, the difference in charge and discharge times would be significantly reduced. Charge times would be shorter, while discharge times would increase, due to decreased heat loss. At the lowest flow rate, charge and discharge times might approach ten hours.

In a solar energy system with one day (around 16 hours) storage, there are approximately 6 hours in which to charge storage. During charging, inlet air temperatures considerably higher than 67.5°C may be expected on a sunny day, and during discharge, inlet temperatures well below 32.5°C would always prevail. These factors would decrease charge and discharge times well below 10 hours. If the requisite 6 hour charging time were still not achieved, one could increase air flow rate, decrease diameter of the PCM capsule, or use a PCM with lower melting point.

Figure 3

OUTLET AIR TEMPERATURE vs TIME

Figure 4

OUTLET AIR TEMPERATURE vs TIME

Table II shows the reduced data from testing of the sub-scale thermal battery. Heat loss through the insulation is greatly reduced from that experienced with magnesium nitrate hexahydrate, due to the lower operating temperature. The energy balance is within acceptable limits indicating the accuracy of the experiments.

The relationships between test time, pressure drop across the thermal battery, and air flow rate through the battery become apparent in Figures 5 and 6. At the target pressure drop of 0.25 inches of water, 9-10 hours is required for charging. If needed, this could be shortened by: 1) reducing the number of containers in the direction of air flow (presently 25), 2) increasing the void fraction in the storage bed, or 3) decreasing the container diameter. The first alternative would reduce the air velocity, and thus the film transfer coefficient, decreasing the capacity of the thermal battery. The second would increase the size of the storage unit, increasing hardware and space costs, and the third would increase the encapsulant cost.

Theoretical charge and discharge capacities (TCC and TDC) were calculated for the storage unit. Pertinent information and results are listed in Table III. Values of TCC and TDC vary slightly because of differences in the specific heat between the liquid and solid state of the phase change material. Both differ from the theoretical storage capacity of ASHRAE (94-77), TSC, in that the initial inlet temperatures and final outlet temperatures are used in the calculation instead of the initial and final inlet temperatures.

Test Results on Magnesium Nitrate Hexahydrate/Ammonium Nitrate Eutectic Encapsulated in R-2 Retort Film Pouches

	1=Charge	2=Discharge
Air Heat Capacity ($\frac{\text{KJ}}{\text{Kg}^\circ\text{C}}$)	1.027	1.015
Density (Kg/M^3)	1.009	1.137

$$\% \text{ Dev.} = \frac{(Q_{\text{in}} - \text{Loss}) - (Q_{\text{out}} + \text{Loss})}{(Q_{\text{in}} - \text{Loss})} \times 100$$

Figure 5

CHARGE (DISCHARGE) TIME vs AIR FLOW RATE

Figure 6

STORAGE UNIT PRESSURE UNIT vs AIR FLOW RATE

TABLE III

Theoretical Charge and Discharge Capacities

Mg(NO₃)₂·6H₂O/NH₄NO₃ Eutectic

Fusion Temp., °C	50
Heat of Fusion, Cal/g	31
Specific Heat, Cal/g°C	0.58 (liquid)
	0.48 (solid)
Total Weight, Kg	86

Steel (storage unit structure
including support racks)

Specific Heat, Cal/g°C	0.10
Total Weight, Kg	162

Temperature Range (approx.), °C	Charging	Discharging
High	58.75	67.50
Low	32.50	41.25
Theoretical Capacity, KJ	17,795	18,110
	(TCC)	(TCD)

The theoretical and measured capacities for charge and discharge are plotted against air flow rate in Figure 7. Figure 8 shows the percent of theoretical capacity attained. The actual capacity is somewhat low in comparison with the previous experiments using $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ and $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$. This is because the spacing between the racks holding the encapsulated PCM is greater than required for minimizing the pressure drop. A closer spacing would have made it difficult to insert the pouches in the racks. The system charged to near full capacity, but discharge capacity dropped off significantly, primarily due to heat loss during discharge.

The poor surface to volume ratio of the cabinet of the subscale thermal battery, accentuated by the high void fraction, 0.82, promotes excess heat loss. A full scale system with lower surface to volume ratio and less void volume should show a higher discharge capacity if adequate insulation were used.

As mentioned previously, reduction of the number of rows in the direction of air flow might have a negative effect on storage capacity. However, this system used roughly half the number of rows in the direction of air flow, but achieved results essentially equivalent to earlier tests with calcium chloride hexahydrate and magnesium nitrate hexahydrate. This probably is due to the extra heat transfer surface per unit weight of PCM. The composite film pouches (ignoring the seals) have a surface to volume ratio of 95 M^{-1} , versus 61 M^{-1} for the aerosol cans used with $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and 66 M^{-1} for the polyethylene bottles used with $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$. Also, the configuration of this system is significantly different from the array of cylinders on an equilateral pitch used in the two earlier experiments. The relatively flat faces of the pouches, perpendicular to air flow, may have introduced significant turbulence.

Figure 7

CAPACITY (ENERGY) vs AIR FLOW RATE

Figure 8

% CAPACITY (ENERGY) vs AIR FLOW RATE

The next configuration to be tested may resolve this question. It will also employ 25 containers in the direction of air flow, using the same racks, but the containers will be substantially similar to the cylinders used with calcium chloride hexahydrate and magnesium nitrate hexahydrate.

IV. UTILIZATION ACTIVITY

Calcium chloride hexahydrate is ready for demonstration testing in thermal batteries. Potential applications include: tempered water heat pump systems, solar heated greenhouses, retrofit solar heating systems using nighttime setback thermostats, and passive solar heating systems. Macroencapsulated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ is appropriate for all these applications, except tempered water heat pump systems. Several potential experiments are being considered.

We have filled 2160 aerosol cans with 3850 pounds $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$, to be supplied to Pennsylvania State University. They will test these in a forced air thermal battery in a 184 ft² greenhouse with 400 ft² added solar collector.

We have contacted personnel at the Solar Room Co., Taos, NM, who are interested in testing encapsulated $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ in their hybrid passive test rooms.

Dr. F. C. Wessling, Albuzuerque, NM, has expressed interest in testing plastic pipes filled with $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ in a direct solar gain configuration.

We have discussed with Stan Moore of Los Alamos Scientific Laboratory supplying 1700 pounds of Dow Experimental Storage Material XFS-43076, a CaCl_2 product with additives, to be used to study $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ in a direct solar gain storage application.

V. PLANS

1. Initiate testing of NH_4Br /urea eutectic, encapsulated in polypropylene bottles, in the storage test device.
2. Continue cycling tests on PCM-encapsulant candidates.
3. Update computer model to fit PCM storage test results.
4. Update economic analysis of thermal storage unit design.
5. Evaluate storage unit designs in total solar systems using TRNSYS.
6. Design residence-sized heat storage sub-system.

VI. CONCLUSIONS

Testing has been completed on $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}/\text{NH}_4\text{NO}_3$ eutectics encapsulated in R-2 Retort Film pouches. Good values were gotten for charge and discharge capacities, and a good energy balance was obtained. Measured capacities averaged 97% of theory for charging and 89% for discharge.

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